PREPARATION OF STANDARDS FOR AQUEOUS TRACER CONCENTRATION MEASUREMENTS

PROCEDURE ID: YMP-LBNL-TIP/AFT 3.0	REV. 0, MOD. 0	EFFECTIVE: 9/4/98

1. PURPOSE

This Technical Implementing Procedure (TIP) describes the preparation of standard solutions for measuring aqueous concentrations of tracers that are used for the Yucca Mountain Site Characterization Project (YMP) at Lawrence Berkeley National Laboratory (LBNL).

2. SCOPE

This procedure applies to all LBNL personnel (or contractor personnel following LBNL procedures) involved in the preparation of standard solutions for measuring aqueous tracer concentrations. This TIP describes the preparation of standards for any analytical procedure involving tracer concentration measurements in which the measurement technique relies on relating the measurement of an unknown to the measurements of a standard or a set of standards. The analytical measurements themselves will be covered by individual TIP's that address the analytical procedure. This procedure also applies to the handling and storage of standards. Prior to conducting work described in Section 3 of this procedure, personnel performing measurements require training to this procedure.

If this procedure cannot be implemented as written, YMP-LBNL personnel should notify the responsible Principal Investigator (PI). If it is determined that a portion of the work cannot be accomplished as described in this TIP, or would produce undesirable results, that portion of the work shall be stopped and not resumed until this procedure is modified per YMP-LBNL-QIP-5.2, *Preparing Quality & Technical Implementing Procedures*.

If the responsible PI determines that a modification or a revision to the TIP would cause an unreasonable delay in proceeding with the task, then an expedited change to the procedure, including documentation of deviation from the approved procedure, can be made according to YMP-LBNL-QIP-5.2. Such changes are subject to review, usually after the task has proceeded, and thus work performed under TIPs with expedited changes is done at risk of future invalidation.

Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used. When this procedure becomes obsolete or superseded, it must be destroyed or marked "superseded" to ensure that this document is not used to perform work.

3. PROCEDURE

3.1 Principle

Most analytical determinations of aqueous tracer concentrations involve measurements that must be related to measurements of known concentrations of the tracers in solution (i.e., the measurements are relative, not absolute, and therefore they must be compared to measurements of known concentrations). The solutions containing the known concentrations are called standards. Starting solution (s) are obtained by weighing an amount of tracer chemical and dissolving it in a known amount of water. A series of dilutions of the starting tracer solution (s) are prepared to obtain a set of standards that span the expected range of concentrations in the unknowns.

Information about the purity of tracer chemicals for making the standards will be requested of the commercial vendors or other available sources. Lot analysis, if available from the sources, will also be requested.

Generally, reagent water, produced from the laboratory water purification system, is used to make solutions unless the investigation requires the use of alternate water (e.g., J13 well water from Yucca Mountain, synthetic water to simulate J13 well water composition, construction water from Exploratory Studies Facility, etc).

In tracer tests where chemicals of unknown purity might be used (e.g., bulk order for large amounts of chemicals required in field tracer experiments), the tracer concentrations in samples are determined by the standards prepared by this TIP. Carrier solution for the tracer tests are sampled and analyzed to assess any interference or background concentrations that will be present in the samples to be analyzed.

3.2 Equipment and Hardware/Software

The equipment required for this TIP includes balances for accurate determination of masses of reagents and volumetric glassware, including pipettes and volumetric flasks. In certain situations, other equipment, such as stirrers and temperature control devices (e.g., hot plates) may also be required. Any balances that are used shall be calibrated and controlled in accordance with YMP-LBNL-TIP/AFT 1.0, *Balance Calibration*.

The accuracy of volumetric flasks, pipettes and pipettors are verified by weighing water quantities delivered (pipettes, pipettors) or contained (flasks) using a calibrated balance. Temperature of water is measured with a calibrated thermometer. Density of water is obtained using appropriate tables of water density vs. temperature (e.g., Lide, 1991). The actual volume is calculated from the measured mass of water divided by its density at the measured temperature. The verification shall be documented in a scientific notebook. Flasks, pipettes and pipettors that do not meet the accuracy (e.g., within 1% of its nominal volume) shall be discarded.

Labware used to make the standard solutions shall be washed in the appropriate cleaning solution, rinsed three times with the tap water, rinsed three times with the reagent water, and air-dried at the room temperature.

3.2.1 Equipment Malfunctions

Malfunctions of any of the equipment described above will be either immediately obvious to the user or will result in the inability to conduct the procedures described in this TIP.

3.2.2 Safety Considerations

Safety considerations will depend on the chemical nature of the constituents in the solutions being prepared. Material safety data sheets (MSDSs) should be consulted to determine whether special protective clothing and/or eye protection are required. Hazardous chemical wastes shall be properly disposed of.

3.2.3 Special Handling

Handling of all equipment associated with this TIP should be done in accordance with manufacturer or vendor's guidelines. Special handling of equipment or hardware should be considered on a case-by-case basis as the need arises. Any special handling shall be documented in a scientific notebook in accordance with YMP-LBNL-QIP-SIII.0, *Scientific Investigation*.

3.3 Preparatory Verification

3.3.1 Hold Points

N/A

3.3.2 Calibration

Balances and thermometers must be controlled pursuant to YMP-LBNL-QIP-12.0, *Control and Calibration of Measuring Equipment*, and balances shall be calibrated in accordance to YMP-LBNL-TIP/AFT 1.0, *Balance Calibration*.

3.3.3 Environmental Conditions

When standards are prepared, good laboratory practices shall be followed to maintain a clean laboratory environment to avoid contamination of the standards. The standards shall be prepared at room temperature to minimize volumetric errors (unless the volumetric glassware requires another temperature).

3.4 Control of Samples

Standard solutions are to be identified and controlled as "samples" in accordance with YMP-LBNL-QIP-SII.0, *Documenting Sample Control*. Standards can be stored in any airtight container that does not compromise them by introducing contaminants that could interfere with the analyses. Glass and plastic bottles are generally acceptable, although the choice of labware shall consider the potential sorption of tracers to container walls. If sorption is unavoidable, special procedures shall be followed to desorb the tracers prior to analyses (e.g., acidification). These procedures shall be documented in a scientific notebook. If there is any question about sorption to container walls, batch sorption experiments shall be conducted (and documented in a laboratory notebook) using the tracers and labware in question. The stability of the tracers shall be considered when storing them. For example, if an UV light sensitive tracer is used (e.g., a fluorescent dye such as fluorescein), the samples shall be stored in darkness or in UV opaque bottles. Factors such as physico-chemical and biological characteristics of the tracers, storage conditions and liquid properties, shall be taken into account to determine the storage life of the standards. Standards shall generally be analyzed at the same temperature as the unknowns.

3.5 Implementing Procedure

Preparation of standards for aqueous tracer concentration measurements involves the following steps:

3.5.1 Starting Solution (s) for Standards Preparation

A. A small amount of tracer chemical (generally a powder) shall be accurately weighed using a calibrated balance. If the tracer chemical is a solution, the weight can be used to determine a volume (assuming that the density is known). Alternatively, for solutions, it may be necessary to use volumetric glassware to dispense a known volume of the solution.

B. After the appropriate amount of tracer chemical is weighed into appropriate containers (e.g., weighting paper, weighting boat, etc.), the chemicals are transferred into glass beakers.

C. Add water in a volume less than ultimately needed and use a stainless steel spatula to stir and dissolve the chemical. Visually inspect the solution to determine if the chemical is fully dissolved.

D. Transfer the solution into the appropriate size of volumetric flask. Rinse the beaker and spatula at least twice with water to assure the complete transfer of chemical into the volumetric flask, collect the rinsate in the volumetric flask, and add water to the calibration line of the flask.

E. Place the glass stopper on the volumetric flask and mix the solution thoroughly by inverting the flask nominally twenty times while holding the stopper.

F. Record in a scientific notebook all weight readings, size of volumetric flask, and/or any other steps taken to accurately determine the mass or volume of tracer chemical that is used for standards preparation. This material will be

referred to as the "starting solution (s)" in the procedure described below. Record the unique identifier of any measuring and test equipment (e.g., balances, thermometers).

Note: Generally, reagent water, produced from the laboratory water purification system, is used to make solutions unless the investigation requires the use of alternate water (e.g., J13 well water from Yucca Mountain, synthetic water to simulate J13 well water composition, construction water from Exploratory Studies Facility, etc). The PI shall make a determination of what type of water shall be used to prepare standards.

3.5.2 Serial Dilution

A. Standard preparation generally involves preparing a series of dilutions of the starting tracer solution (s) to obtain a set of standards that span the expected range of concentrations in the unknowns. Subsequent dilutions to prepare lower concentration standards can be done in series by pipetting a known volume of each successive standard prepared (starting with the first, most concentrated one) into a volumetric flask and then diluting to the reference line with the water. This procedure, known as "serial dilution", is a widely recognized and accepted method of preparing standards for a wide range of chemical analysis techniques.

B. Record in a scientific notebook steps taken to make serial dilution (i.e., volumes pipetted and size of volumetric flasks), the ambient temperature in the laboratory (if applicable), and any unusual occurrences or situations that could result in errors in the standards concentrations.

C. Prepare a label indicating the chemical's name and concentration, preparation date, preparer's initial, and attach the label to the individual container of standard solutions.

3.6 Data Acquisition and Reduction

With the exception of weight readings on balances and calculations to determine concentrations of standards based on weights and volumes, this TIP does not involve data acquisition or reduction. Data acquisition and reduction associated with analyses of tracers will be covered by separate TIP's that control the operation of the appropriate analytical instruments.

The criteria for acceptance of prepared standards are (1) the steps taken to prepare the standards are properly documented as prescribed in subsection 3.5, (2) any balances and thermometers used were properly calibrated, and (3) volumetric glassware used for all dilutions were verified for volume accuracy. The PI ultimately reviews all data and records associated with standards preparation and determines the acceptability of the standards and the tracer concentration measurements that are conducted using the standards; the review shall be documented in the scientific notebook. The PI may reject standards if it appears that the standards do not follow expected trends when they are analyzed with analytical

equipment (e.g., standards that are supposed to have a factor of 10 difference in concentration give responses that suggest only a factor of 5 difference). The identity of rejected standards and the basis for rejection are recorded in a scientific notebook.

3.7 Potential Sources of Error and Uncertainty

Potential sources of error and uncertainty associated with the implementation of this TIP include (but are not necessarily limited to) the following:

- Inaccurate weight or volume measurements,
- Preparing standards at a significantly different temperature than the volumetric glassware is intended for,
- Use of dirty labware that contains contamination or significant levels of the constituents that interfere with the analyses of the target tracer, and
- Improper storage of standards such that they sorb to container walls, degrade or are otherwise compromised.

If a problem arises during standards preparation, handling, or storage that can be considered a potential source of error or uncertainty when conducting tracer analyses, the PI or staff member shall document the problem in a scientific notebook. The PI or staff member can then later assess the impact of the source of error or uncertainty when analyzing the measured tracer concentrations.

3.8 Acceptance Criteria

Proper implementation of this procedure and submittal of the records constitutes the acceptance criteria for this procedure.

4. RECORDS

4.1 Lifetime

Records generated as a result of this TIP are entries in scientific notebooks or attachments to such notebooks.

4.2 Non-Permanent

None

4.3 Controlled Documents

Technical Implementing Procedure

4.4 Records Center Documents

Records associated with this procedure shall be submitted to Records Processing Center in accordance with AP-17.1Q, *Record Source Responsibility for Inclusionary Records*.

5. RESPONSIBILITIES

5.1 The **Project Manager** is responsible for final approval of the new, revised or modified TIP and for final approval of the rescission of the TIP.

5.2 The **EA Manager** is responsible for approving the new, revised or modified TIP, and for the rescission of the TIP.

5.3 The **OQA Representative** is responsible for reviewing and concurring with the TIP.

5.4 The **Principal Investigator (PI)** is responsible for assuring full compliance with this procedure and of overseeing and coordinating the preparation, review, distribution, revision, and recommending rescission of the TIP, and for the related training of personnel.

5.5 **Staff Members** involved in the preparation or review of procedures are responsible for following this procedure and turning over related documentation to the Records Coordinator for submittal to the Records Processing Center in accordance with AP-17.1Q.

5.6 **Document Control Staff** are responsible for providing the controlled distribution of the TIP and modifications to it.

6. ACRONYMS AND DEFINITIONS

6.1 Acronyms

LBNL Lawrence Berkeley National Laboratory

MSDS Material Safety Data Sheet

TIP Technical Implementing Procedure

YMP Yucca Mountain Site Characterization Project

6.2 Definitions

Standard A standard is a solution containing an accurately known concentration of a tracer or a set of tracers that have been used in tracer tests. The standard (or, more generally, a series of standards) will be used to calibrate or obtain a calibration curve for analytical instrumentation that is used to determine the concentration of tracers in samples.

Staff Member Any scientist, engineer, research or technical associate, technician, or student research assistant performing quality-affecting work for YMP-LBNL.

Technical Implementing Procedure Each TIP describes YMP-LBNL technical and/or scientific tasks that (1) are repetitive, (2) are standardized, and (3) could return different results should deviation from the sequence of steps occur. TIPs are written when such tasks are sufficiently complex to warrant a formal procedure.

7. REFERENCES

AP-17.1Q. *Record Source Responsibility for Inclusionary Records*.

YMP-LBNL-QIP-5.2 *Preparing Quality & Technical Implementing Procedure.*

YMP-LBNL-QIP-12.0. *Control and Calibration of Measuring Equipment.*

YMP-LBNL-QIP-SII.0. *Documenting Sample Control*.

YMP-LBNL-QIP-SIII.0. *Scientific Investigation*.

YMP-LBNL-TIP/AFT 1.0. *Balance Calibration.*

Lide, D. R. (Editor-in-Chief). 1991. *CRC Handbook of Chemistry and Physics*, 71th edition, CRC Press, Inc. Boca Raton, FL.

8. ATTACHMENTS

None

9. REVISION HISTORY

9/4/98 - Revision 0, Modification 0:

Initial issue of this procedure.

10. APPROVAL

Preparer: Qinhong (Max) Hu	Date:
Technical Review: Tim Kneafsey	Date:
Technical Review: Scott Mountford	Date:
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OQA Concurrence: Stephen Harris	Date:
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